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AN INVESTIGATION OF THE CRYOGENIC MECHANICAL PROPERTIES OF LOW THERMAL-EXPANSION SUPERALLOYS

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ABSTRACT

Four Fe-based superalloys, JBK-75, Incoloy 903, Incoloy 905, and Incoloy 909 were evaluated as tube materials for ICCS Nb_3Sn superconductors. Evaluation consisted of 4-K tensile and elastic-plastic fracture-toughness testing, and a microstructural characterization of unwelded and autogenously gas-tungsten-arc welded sheet given a simulated postweld processing treatment of 15% cold reduction by rolling followed by a Nb_3Sn-reaction heat treatment of 96 hours at 700°C plus 48 hours at 730°C. Results indicate that JBK-75 and Incoloy 903 showed satisfactory combinations of strength and toughness for ICCS tube use requiring long Nb_3Sn-reaction heat treatments. Incoloy 905 welds and 909 showed unacceptable fracture toughness. Results are discussed in terms of microstructural changes caused by the extended Nb_3Sn-reaction heat treatment.

INTRODUCTION

Many fusion coil designs[1-3] use unalloyed or alloyed Nb_3Sn superconductor, surrounded by a thin-walled tube, and cooled by the forced flow of l He inside this tube; the so-called "internally cooled coiled superconductor" or ICCS. This report describes the investigation of the suitability of four alloys proposed for use as the tube.

The high-strain sensitivity of A15 conductors[4] requires that the conductor be fully fabricated prior to reaction heat treatment. Fabrication includes wrapping with the tube alloy, seam welding, and swaging to final size. The tube alloy sees the high-temperature heat treatment that forms Nb_3Sn in the conductor. Minimizing thermal-expansion mismatch between the Nb_3Sn conductor and tube is beneficial, and use of alloys with controlled thermal expansion can substantially increase the current-carrying capacity of short lengths of ICCS conductors.[5]

Tube-alloy performance targets in this program are those set forth in the Vail Workshop[6] in October 1984: a 4-K yield strength (σ_y) of at least 700 MPa, a 4-K fracture toughness (K_{IC}) of at least 100 MPa $\sqrt{m}$, and a tensile elongation of at least 10%. In this investigation, the 4-K K_{IC} and tensile properties of four candidate alloys were determined on unwelded sheet and autogenous (without filler material being added) gas-tungsten-arc (GTA) welds after a simulated Nb_3Sn-reaction heat treatment of 96 hours at 700°C plus 48 hours at 730°C. Work performed by Wu et al.[7] showed that this treatment yielded the maximum critical current density for internal bronze-process Nb_3Sn superconductor.

Four alloys were selected for evaluation. The first, JBK-75, is the same precipitation-hardening stainless steel used for the tube for the Westinghouse Large Coil.[8] The three remaining alloys (Incoloy 903, 905, and 909) are controlled thermal-expansion alloys, with compositions given in Table I. All four alloys harden by the precipitation of an ordered intermetallic phase of the "Ni_3X" type, where "X" may be Ti, Al, or Nb, either alone or in combination.

PROCEDURE

The starting material was 1.54- to 1.77-mm-thick sheets. Sheets were sealed in stainless steel bags and solution treated in air for one hour at 1253 K. Following descaling, weld specimens were prepared using the GTA process. All welds were full penetration "bead-on-plate" welds.

Metallographic specimens were sealed in stainless steel bags and heat treated in air at temperatures from 923 to 1073 K for up to 500 hours. Temperatures were controlled to ± 2 K, as determined by a chromel-alumel thermocouple. Specimens for mechanical testing were aged using the previously described heat treatment.[7]

Aging curves were generated using microhardness measurements obtained from metallographic specimens. Mechanical test specimens were described in Ref. 9. The K_{IC} was determined using the multiple-unloading-compliance J-integral method of ASTM E-813[10] modified for testing at 4 K.[11] Tensile tests were run at a crosshead speed of 1.27 mm/min in a "hard" machine and testing conformed to procedures set forth in ASTM E-8[12], modified for testing at 4 K.[13] Specimens were oriented such that the applied loads were taken both along and transverse to the rolling/welding direction .

RESULTS

Mechanical Testing

Results appear in Table II. The average ultimate tensile strengths (σ_u), 0.2% offset yield strengths, and elongations to failure (e_f) of the four alloys are given. The 4-K σ_y of all unwelded and GTA-welded sheet surpass the 700-MPa goal. Elongation values of Incoloy 903, 905, and JBK-75 sheet exceed the 10.0% requirement. Elongation values of Incoloy 909 sheet were unacceptably low, with values of 4.6% (L) and 7.0% (T). Elongation values of Incoloy 905 welds tested in the T direction and JBK-75 welds tested in the L direction exceeded the 10.0% requirement. However, e_f values for Incoloy 903 welds tested in both directions and JBK-75 welds tested in the T direction were between 9.0 and 10.0%. Incoloy 905 welds tested in the L direction and Incoloy 909 welds tested in both directions fell well below the 10.0% requirement, being 7.4%, 4.0%, and 3.5%, respectively. Incoloy 909 had a weld e_f of 3.75% while Incoloy 905 and 903 welds showed e_f of 7.4 to 14.2% and 9.1 to 9.3%, respectively.

Notched tensile data at a stress concentration of 4 are summarized in Table II. Incoloy 903 and JBK-75 had ratios of notched tensile strength (σ_{un}) to unnotched tensile strength, or "NTR", no lower than 0.81. Welded Incoloy 905 had a NTR ratio of 0.71. Incoloy 909 had significantly lower NTR values, 0.65 to 0.73 for sheet and 0.55 to 0.56 for welds.

Fracture toughnesses of all alloys are given in Table II. Incoloy 909 sheet and welds have unacceptably low K_{IC} of 69.7 MPa $\sqrt{m}$, and 38.9 to 39.5 MPa $\sqrt{m}$, respectively. Incoloy 905 welds tested in the L direction had a K_{IC} of 81.5 MPa $\sqrt{m}$, but this value rose to 136.8 MPa $\sqrt{m}$ when tested in the T direction.

The 4-K K_{IC} values showed a qualitative correlation to NTR and ratio of σ_{un}/σ_y, or "notched yield ratio", NYR. Those materials with high K_{IC} displayed high NTR and NYR values, while those with low K_{IC} showed low NTR and NYR ratios. For example, Incoloy 909, which has a weld K_{IC} of about 39 MPa $\sqrt{m}$, has an NTR of about 0.55.

Modulus of elasticity (E) values for the four alloys are in Table II. These values were determined from the elastic-strain region of the tensile stress-strain curves. Data are reported for E values obtained for sheet L and T orientations and weld L orientations. Note that the two Co-free alloys (JBK-75 and Incoloy 905) have significantly higher (in excess of 170 GPa) E values than Incoloy 903 and 909, which have E values of 138 GPa to 156 GPa and 141 GPa to 174 GPa, respectively.

MICROSTRUCTURAL CHANGES

For tensile and K_{IC} rupture surfaces of sheet, microstructural observations are summarized in Table III. Incoloy 903 showed ductile rupture. JBK-75 showed ductile rupture in grain interiors with embrittlement of grain boundaries. Incoloy 905 showed a mixed-mode failure with regions of transgranular cleavage and of intergranular fracture. Incoloy 909 is embrittled, with a rough, intergranular, fracture surface.

Microstructural observations on fracture surfaces of heat-treated welds are summarized in Table III. Incoloy 903 and JBK-75 showed failures that are mostly ductile. A dendritic weld microstructure was evident, indicating that some embrittlement occurred at dendrite interfaces. Incoloy 903 showed evidence of the fracture following the columnar weld-metal grain-structure. Incoloy 905 and 909 showed pronounced evidence of separation both along weld-metal-grain and dendrite boundaries.

JBK-75 formed grain-boundary phases by a cellular precipitation mechanism.[14] The cellular product was seen in the weld at aging times of less than 8 hours. Continued aging resulted in rapid coarsening of the cellular product in the weld and decoration of sheet grain boundaries by the cellular product. The common overaging product in Fe-Cr-Ni superalloys is Ni_3Ti eta phase[9] which forms by a cellular mechanism, and is probably responsible for the weld and sheet grain-boundary decoration in JBK-75.

Fully reacted Incoloy 903 weld and sheet showed discontinuous precipitation along grain boundaries and a coarse intragranular precipitate. Semi-quantitative SEM-EDS analysis showed that the intergranular precipitates are slightly enriched in Nb and Ti with respect to the matrix, indicating that these precipitates are of the $Ni_3(Nb,Ti)$ type.

Fully reacted Incoloy 905 sheet showed serrated grain boundaries resulting from grain-boundary migration caused by growth of the coarse, intergranular, precipitate plates. Incoloy 905 welds showed intragranular precipitates. Incoloy 909 sheet and welds showed heavy, small, intergranular precipitation early in aging. SEM-EDS analysis showed enrichment with Nb and Ti. These precipitates are probably an intermetallic averaging product, such as $Ni_3(Nb, Ti)$.

Weld and sheet specimens of these alloys showed a difference in aging response. The welds age more slowly and to lower peak-hardness values than does sheet.[9]

DISCUSSION

Susceptibility of Fe-base superalloys to formation of embrittling precipitates is complex, and depends on both processing history and composition. Holding other factors constant, the alloy with the higher concentration of hardening elements such as Ti, Al, Nb, Ta, and C, will be most likely to form unwanted phases. These assumptions provide guidelines for material selection and understanding of microstructural changes.

Low K_{IC} and rapid grain-boundary precipitation in Incoloy 909 is expected, since the combined Al, Ti and Nb content of this alloy is nominally 6.6 wt%. The high Nb/Ti ratio favors the substitution of Nb for Ti in γ', or the possible formation of γ''.[15] Substitution of Nb for Ti increases lattice mismatch and the strain energy that drives the formation of equilibrium phases.

Incoloy 905 contains large amounts of elements that participate in hardening-phase formation (6.33 wt%). Replacement of Co with Ni in this alloy affects the precipitate morphology. Overaging products, rather than forming as grain-boundary precipitates, form as intragranular Widmanstatten precipitates that contribute to transgranular fracture and reduction of K_{IC}.

Both JBK-75 and Incoloy 903 contain lesser reduction of the hardening elements: 3.4 and 5.3 wt%, respectively. Also significant is the lower Nb/Ti ratio in Incoloy 903 as compared to other Incoloys, resulting in alloys that, while showing grain-boundary precipitation, do not do so to an extent that K_{IC} is compromised. Welds in JBK-75 are sensitive to formation of a cellular phase mixture at grain and dendrite interfaces, due to segregation of Ti to dendrite interfaces during weld solidification.[9] Hardness mismatches during isothermal heat treatments of these alloys are caused by this Ti segregation, which forms "precipitate-free zones" (pfz) in welds. Multistep aging treatments reduce the extent of pfz. Similar σ_y for welds and sheets in this investigation results from beneficial effects of the long aging at 973 K.[9]

Based on K_{IC} and σ_y requirements, Incoloy 903 and JBK-75 meet the specified targets. The low e_f of Incoloy 903 weld is below the needed 10%. Owing to the weld's high K_{IC}, this may not be a major concern. Incoloy 905 and 909 have good σ_u and σ_y, but insufficient K_{IC}, particularly in their welds.

Incoloy 903 and 909 have low E values, about 156 GPa. Incoloy 905 and JBK-75 have values of 204 GPa and 238 GPa, respectively. If the tube is a load-bearing member, designers must account for differences in moduli of tube alloys. Use of a higher-E alloy, such as JBK-75, may allow reductions in tube-wall thicknesses and a higher fraction of superconductor in the coil pack. However this advantage must be weighed against effects of thermally induced strains caused by the higher thermal-contraction coefficient of JBK-75.

Alloys with large amounts of Co (Incoloy 903 and Incoloy 909) had lowest E values. It is assumed that replacement of Ni and Fe with Co is responsible for E changes. If this is true, development of E-composition correlations based on published information may provide a means for planning a focused alloy-design program to produce an alloy with both high E and low thermal-expansion coefficient.

CONCLUSIONS

1. All alloys formed extensive coarse precipitates during a long (973 K:96 hrs plus 1003 K:48 hrs) Nb_3Sn-reaction heat treatment. Incoloy 909 and 905 sheet and GTA welds showed heavy intergranular and intragranular precipitation. JBK-75 welds showed extensive formation of an Ni_3Ti-based phase that did not adversely affect K_{IC}. Both JBK-75 sheet and Incoloy 903 sheet and welds showed much less sensitivity to overaging than did Incoloy 905 and 909.

2. Yield strengths of all alloys are above the 700-MPa requirement. However, Incoloy 905 welds and Incoloy 909 welds and sheet had K_{IC} values that are well below the 100 MPa $\sqrt{m}$ target. Intergranular failure and reduced K_{IC} resulted from heavy precipitation of brittle equilibrium phases that occurred intragranularly in Incoloy 905 and at the dendrite and grain boundaries of Incoloy 909.

3. Tensile elongation is acceptable in JBK-75 weld and sheet. Incoloy 903 and 905 display satisfactory e_f in sheet but marginal e_f in welds. Incoloy 909 had poor e_f in both weld and sheet.

4. The E values of Incoloy 903 and 909 are considerably lower than Incoloy 905 and JBK-75, possibly due to the replacement of Ni and Fe with Co in these alloys.

RECOMMENDATIONS

Based upon 4-K mechanical properties, Incoloy 903 and JBK-75 are acceptable for ICCS conductor-tube alloy usage in applications requiring extended Nb_3Sn heat treatments. The E value of Incoloy 903 is low and its effect on any design must be examined. Incoloy 905 and 909 should be excluded from consideration on the basis of low K_{IC} values.

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Table I

Compositions, weight percent

Alloy	Fe	Cr	Ni	Co	Nb	Ti	Al	Mo	Mn	C	Si
JBK-75	Bal	15.0	29.0	----	----	2.15	1.25	1.25	0.05	0.016	--
1-903	Bal	----	38.0	15.0	3.0	1.4	0.9	0.5	0.2	0.03	--
1-909	Bal	----	38.2	13.0	4.7	1.5	0.4	0.5	0.2	0.03	0.25
1-905	Bal	----	48.9	0.33	4.7	1.59	0.04	----	0.05	0.01	--

Table II. Mechanical Properties of Candidate ICCS Sheath Alloys and Their Autogeneous GTA Welds at 4.2 K

Alloy	Condition	Orientation	Elastic modulus, GPa	Ultimate strength, MPa	Yield strength, MPa	Elongation in 12.7 mm, %	Plane-strain fracture toughness MPA $\sqrt{m}$	NTR[c]	NYR[d]
Incoloy 903	Unwelded	Longitudinal[a]	138.6	1530	1016	28.9	165.4	0.97	1.46
		Transverse[b]	156.2	1611	1184	27.9	110.1	0.93	1.28
	Welded	Longitudinal	151.5	1499	1139	9.1	127.4	0.83	1.09
		Transverse	——	1439	1070	9.3	150.9	0.88	1.19
Incoloy 905	Unwelded	Longitudinal	171.4	1453	906	11.1	133.0	0.80	1.20
		Transverse	215.7	1527	1024	15.8	140.5	0.85	1.26
	Welded	Longitudinal	195.6	1472	942	7.4	81.5	——	——
		Transverse	——	1502	984	14.2	136.8	0.71	1.02
Incoloy 909	Unwelded	Longitudinal	174.2	1432	1115	4.6	——	0.73	0.94
		Transverse	161.9	1606	1236	7.0	69.7	0.65	0.85
	Welded	Longitudinal	140.9	1506	1119	4.0	39.5	0.55	0.74
		Transverse	——	1454	1084	3.5	38.9	0.56	0.74
JBK-75	Unwelded	Longitudinal	232.7	1884	1398	34.2	108.3	0.93	1.26
		Transverse	274.3	1710	1238	30.0	——	0.96	1.33
	Welded	Longitudinal	243.9	1855	1295	17.5	148.8	0.85	1.26
		Transverse	——	1831	1415	9.7	133.0	0.81	1.06

[a] Longitudinal - parallel to rolling direction of sheet in direction of welding.
[b] Transverse - perpendicular to rolling direction of sheet in direction of welding.
[c] Ratio of notched to unnotched tensile strength at a stress concentration of 4.0.
[d] Ratio of notched to unnotched yield strength at a stress concentration of 4.0.

Table III

Summary of Microstructural Observations on Fully Reacted Alloys

	Incoloy 903		Incoloy 905		Incoloy 909		JBK 75	
Sampling location	Base metal	Weld metal	Base metal	Weld metal	Base metal	Weld metal	Base metal	Weld metal
Fracture surfaces	Ductile rupture	Ductile rupture, separation along dendrites	Cleavage & intergranular fracture	Separation along dendrites	Intergranular fracture	Separation along dendrites	Ductile rupture plus intergranular fracture	Ductile rupture separation along dendrites
General Microstructure	Austenite, discontinuous grain-boundary phase, coarse intragranular precipitate		Coarse, intergranular precipitate, serrated grain boundaries		Semicontinuous grain-boundary phase, fine intragranular precipitate		Austenite plus cellular phases growing from grain boundaries	